Plasma cleaning system ATTO

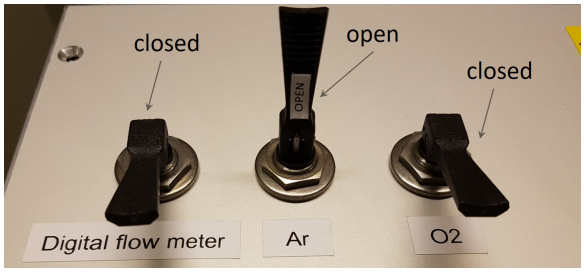
Version: May 9, 2023

OPERATION

1. Turn ON the vacuum pump. The pump should be ON during the plasma treatment process. You turn pump OFF when treated all your samples. The PUMP button on the device is connecting or disconnecting vacuum by a magnetic valve and not turning pump ON/OFF.

2. Turn ON the main switch of the plasma cleaner.

3. Select your processing gas; open one of the three following valves; Ar, O2 or the Digital flow meter. The valves Ar or O2 are used when gas feeded by the needle flow controllers. Digital flow meter when the flow controlled by computer. These valves disconnecting the treatment chamber from all other paths even ventilation, therefore the selected valve should be open during ventilation, only close when switching OFF the plasma cleaner.



4. Set the desired plasma power (**Power range 0-50 W, Frequency 13.56 MHz**). Use the potentiometer marked by POWER to set the power as described in figure. Plasma ignition can be difficult when power is below 20 W.


99 % power


10% power

5. Set the desired processing time (plasma treatment) using the potentiometer marked by TIMER. Alternativ: set the processing time on maximum and count manually by lab timer.

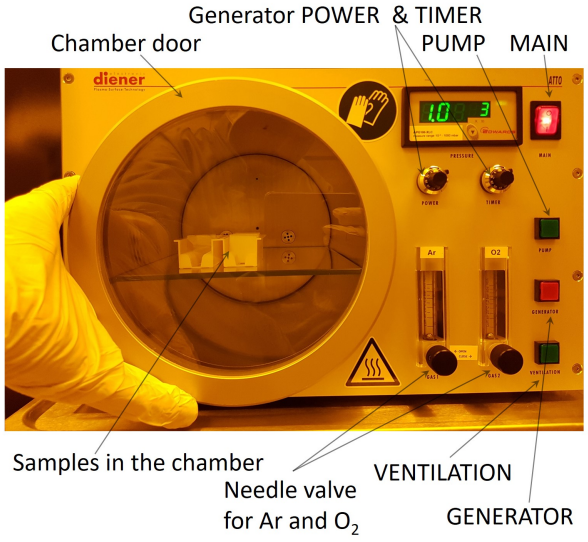

10 % time

1 %	≈ 0.32	min ≈ 19.2	sec
3.1 %	≈ 1	min ≈ 60	sec
10 %	≈ 3.2	min ≈ 192	sec
15.6 %	≈ 5	min ≈ 300	sec
20 %	≈ 6.4	min ≈ 384	sec
31 %	≈ 10	min ≈ 600	sec
47 %	≈ 15	min ≈ 900	sec
50 %	≈ 16	min ≈ 960	sec
63 %	≈ 20	min ≈ 1200	sec
78 %	≈ 25	min ≈ 1500	sec
94 %	≈ 30	min ≈ 1800	sec
100 %	≈ 32	min ≈ 1920	sec

6. Load your samples in the plasma chamber.

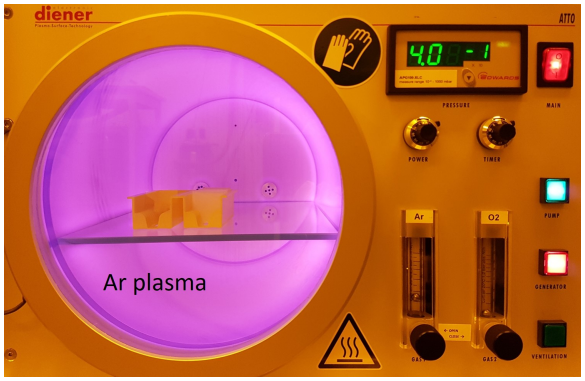
7. Hold the plasma cleaner door against the vacuum chamber.

8. Press PUMP button to start evacuating the chamber. It will take a few minutes to evacuate all air from the plasma cleaner chamber. The vacuum will hold the plasma cleaner door in place, release the door. Keep evacuating the chamber for 4-5 minutes, vakumm will be around 2.5×10^{-1} mbar. The PUMP must be ON during the complete process.



9. If Ar or O2 valves selected (open); slightly open the needle valve and allow the operation gas (Ar or O_2) to enter the plasma cleaner chamber. Keep the gas flow between **20 ml/min** and **30 ml/min** (the same for digital flow meter) during the process and keep eye on the pressure, it should not be higher than 8.0×10^{-1} mbar. At high pressure (above 1 mbar) the plasma will get OFF.

10. When gas flow almost stabilized (after 2-4 minutes, presure about 5×10^{-1} mbar), turn ON the plasma cleaner by pressing GENERATOR button on the front of the plasma cleaner. Plasma will ignite and you will observe a purplish glow in the chamber, this indicates that the plasma has been generated. The colors are different depending on the processing gas.



11. Upon completion of plasma treatment, the generator will turn OFF automatically if the integrated timer is used. If normal lab timer is used, you have to press GENERATOR button to switch OFF the plasma when the time is done.

12. Press PUMP button to disconnect vacuum.

13. Close the needle valve gently (do not turn too tight), to stop gas flow.

14. Hold the chamber door and ventilate the chamber by pressing VENTILATION button and take out your plasma treated samples.

15. Turn OFF the VENTILATION button.

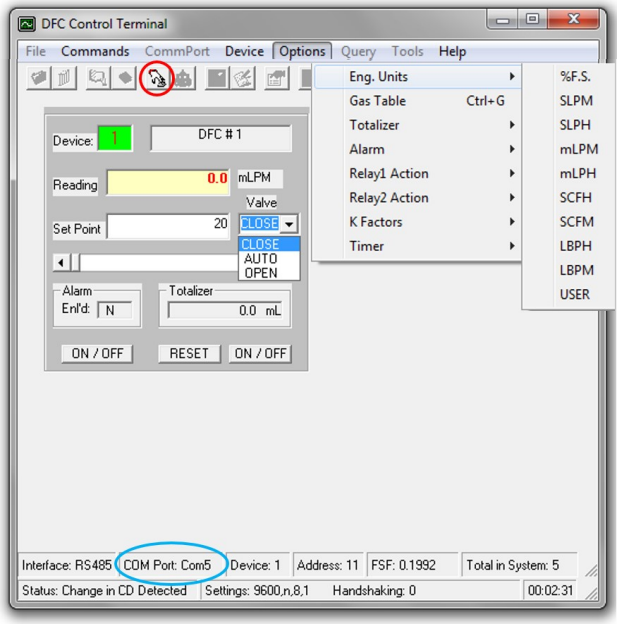
16. When finished: close gas valves, turn OFF the plasma cleaner (MAIN). Clean and organize the working place before leaving the cleanroom.

Treatment complete

Digital flow meter use

1. Connect USB cable of the digital flow meter to a lab computer. Switch ON flow meter and open valve marked "Digital flow meter" on the plasma cleaner (the other two valves Ar and O2 must be closed).

2. Run the software "**DFC Control Terminal**" and press the button marked by red in figure (hand sign).

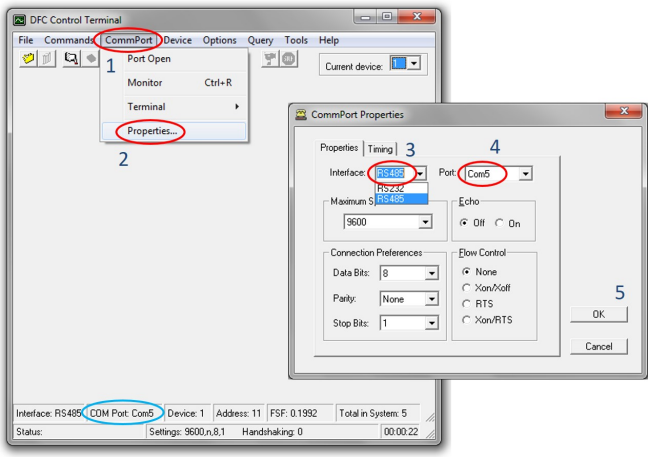


3. Input **Set Point** (20 mLPM) for desired flow (flow range 0 - 200 mL/min), then set Valve on **AUTO**. Set Valve on **CLOSE** when plasma treatment is finished.
Valve on **OPEN** means totally open the valve (high flow, over 100 mL/min, no plasma generation). If other unit than mL/min (mLPM) wanted, go to **Options** ⇒ **Eng.Units** ⇒ choose units from list.

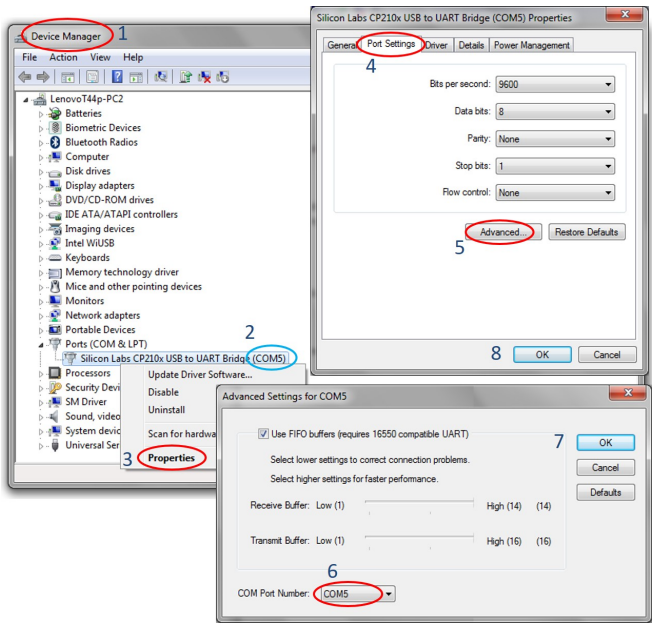
Digital flow meter troubleshooting

Two possible Error message can pop up; "**Invalid port number**" and "**No connection**".
Check connection cables and correct COM port number.

The COM Port should be identical in software and device in Device Manager.
In software: to correct COM port number in software: CommPort ⇒ Properties ⇒ set Port ⇒ OK.
In computer: Start Menu ⇒ Control Panel ⇒ Hardware and Sound ⇒ Device Manager ⇒ Ports (COM & LPT) ⇒ left click on Silicon Labs CP210x USB to UART Bridge ⇒ Properties ⇒ Port Settings ⇒ Advanced ⇒ set COM Port Number ⇒ OK ⇒ OK.
COM5 set as default port during installation.
It is different COM ports setting in different computers. Therefore this should be checked when other computer is used.



Another problem is Interface setting, installation default is RS232. Interface should be set after software installation to **RS485**.
In software: to correct Interface in software: CommPort ⇒ Properties ⇒ set Interface to **RS485** ⇒ OK.
Both COM Port and interface **RS485** are correctly set and still have problems? restart computer.



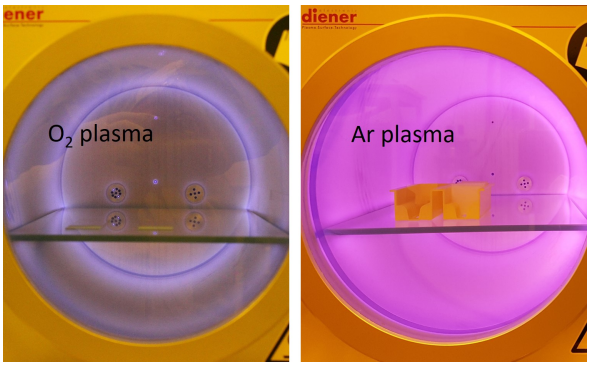
Recommended process parameters

Cleaning: Time= 1-60 min , Power=50-100 % , Gas: O_2, H_2/Ar, O_2/Ar, Pressure= 0.2-0.5 mbar.
Activation: Time= 1-30 min, Power=50-100 % , Gas: O_2, Pressure= 0.2-0.5 mbar. Activation is very unsteady, therefore the samples have short shelf life. Activation is a following process of cleaning.
Gas flow 20-40 mL/min. Look for specific parameters for your material.

Common gases used for plasma treatment

Plasmas are a mixture of reactive species (free radicals, ions, electrons and gas molecules). Plasma treatment is used to clean surfaces and increase/decrease surface energy. Here a gas or gas mixture is ionised by a high frequency voltage to form a reactive gas plasma.
The plasma will interact with the surface in several ways. Contaminants can be removed via ablation, by bombardment with electrons and ions (**Ar plasma**), or through reactions with species (oxidise organic contaminants) in the plasma (**O_2 plasma**). Oxygen and oxygen-containing plasmas are most common for modifying polymer surfaces, it is also a strong disinfectant. O_2 plasma is colorless, you have to use plasma indicators to be able to know if the plasma are generated or not, or use air or argon contaminated O_2.
Inert gas plasmas; He, Ne, O_2, N_2, Ar, H_2, and CF_4 are the most gases used in plasma processing,

although argon is the most common because of its low cost. Unlike oxygen and hydrogen, argon is not chemically reactive, but the comparatively heavy argon ions generated in the plasma exercise a micro-sandblast effect where they hit the surface. Ar plasma has a purplish color.
A gas combination of 25% Ar and 75% O_2 is a very powerful substrate treatment environment.
Hydrogen (H_2) plasma is a powerful reducing agent, it is used to clean metals from oxide films. The hydrogenation of the surface in the plasma essentially increases hydrophobic properties.



Plasma surface activation and grafting

Surface activation by plasma is also referred to as chemical grafting. It never occurs alone, but always occurs during/after a plasma cleaning.
Indeed, in the case of a substrate subjected to a soft secondary plasma which contains reactive species (e.g. oxygen atoms), the effect of those atoms will be twofold: they will react with organic contamination which is present on the substrate surface. Such organic contamination consists, in many cases, of loosely bound hydrocarbons. Both H and C will react with oxygen and will leave the substrate surface in the form of volatile H_2O and CO_2.
Once the surface molecules of a polymer are freed from contamination, they can react with the oxygen atoms which will form carbonyl-, carboxyl- or hydroxyl functional groups on the substrate surface. It is said that the polymer surface has been chemically functionalized.
The effect of grafting carbonyl-groups onto a surface of PP, polyethylene (PE), or polyesters such as polyethyleneterephthalate (PET) or polybutyleneterephthalate (PBT) gives rise to an increase in surface energy to levels higher than 68 mN/m immediately after the plasma treatment.
This effect is, however, not permanent: it has a certain shelf-life. Once the substrate has been removed from the plasma, and depending on the storage conditions, oxygen atoms will be released again from the surface molecules. This will happen slowly over time. After several days or even several months, the original surface energy of the substrate will have returned.
The rate at which this happens depends on the type of substrate: e.g. PP has a fairly good shelf-life of a couple of weeks, whereas silicones show a shelf-life of less than one day. It further depends on the plasma conditions: an intensive plasma treatment will create a higher surface density of functional groups and, as such, the shelf-life will be longer.
Plasma activation is being used in several fabric and nonwoven applications in the textile industry.